A systematic assessment of the diamond trap method for measuring fluid compositions in high-pressure experiments

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ABSTRACT

A variety of experimental techniques have been proposed to measure the composition of aqueous fluids in high-pressure experiments. In particular, the “diamond trap method,” where the fluid is sampled in the pore space of diamond powder and analyzed by laser-ablation ICP-MS after the experiment, has become a popular tool. Here, we carried out several tests to assess the reliability of this method. 

(1) We prepared several capsules loaded with fluid of known composition and analyzed the fluid by laser-ablation ICP-MS, either (a) after drying the diamond trap at ambient condition; (b) after freezing and subsequent freeze-drying; and (c) after freezing and by analyzing a frozen state. Of these methods, the analysis in the frozen state (c) was most accurate, while the results from the other two methods were poorly reproducible, and the averages sometimes deviated from the expected composition by more than a factor of 2. 

(2) We tested the reliability of the diamond trap method by using it to measure mineral solubilities in some well-studied systems at high pressure and high temperature in piston-cylinder runs. In the systems quartz-H2O, forsterite-enstatite-H2O, and albite-H2O, the results from analyzing the diamond trap in a frozen state by laser-ablation ICP-MS generally agreed well with the expected compositions according to literature data. However, in the systems corundum-H2O and rutile-H2O, the data from the analysis of the diamond trap were poorly reproducible and appeared to indicate much higher solubilities than expected. We attribute this not to some unreliability of the analytical method, but instead to the fact that in these systems, minor temperature gradients along the capsule may induce the dissolution and re-precipitation of material during the run, which causes a contamination of the diamond trap by solid phases. 

(3) We carried out several tests on the reliability of the diamond trap to measure fluid compositions and trace element partition coefficients in the eclogite-fluid system at 4 GPa and 800 °C using piston-cylinder experiments. The good agreement between “forward” and “reversed” experiments—with trace elements initially either doped in the solid starting material or the fluid—as well as the independence of partition coefficients on bulk concentrations suggests that the data obtained are reliable in most cases. We also show that the rate of quenching/cooling has little effect on the analytical results, that temperature oscillations during the run can be used to enhance grain growth, and that well-equilibrated samples can be obtained in conventional piston-cylinder runs. Overall, our results suggest that the diamond trap method combined with laser-ablation ICP-MS in frozen state yields reliable results accurate within a factor of two in most cases; however, the precipitation of accessory minerals in the diamond trap during the run may severely affect the data in some systems and may lead to a gross overestimation of fluid concentrations.

Keywords: Fluids, diamond trap, high-pressure experiments, laser-ablation ICP-MS, solubility, fluid/mineral partitioning

INTRODUCTION

Aqueous fluids are important agents of metasomatism in Earth’s mantle, particularly above subduction zones (e.g., Tatsumi 1989; Manning 2004; Kelley and Cottrell 2009; Keppler 2017). Traces of such fluids are sometimes sampled as fluid inclusions in mantle xenoliths and in diamonds (e.g., Kawamoto et al. 2013; Weiss et al. 2015). However, in particular the fluids sampled by diamonds may be the result of extensive fractionation processes, which are not easy to unravel. Constraining the primary composition of mantle fluids therefore requires experimental studies. Unfortunately, methods for the direct withdrawal and analysis of fluids are limited to very low pressures (Potter et al. 1987) and cannot be used under typical mantle P,T conditions. Simply quenching fluids equilibrated with minerals at high P and T and analyzing the quenched fluid at ambient conditions is not likely to yield meaningful results, because in most cases, solutes will precipitate as solid phases even during rapid quenching (e.g., Ryabchikov and Boettcher 1980). Various methods have been proposed to solve this problem. In simple systems, where minerals dissolve congruently, the weight loss of single crystals may allow very accurate solubility measurements, since during quenching, solute will precipitate throughout the fluid, and only a very minor fraction could produce an overgrowth on the original single crystal (e.g., Manning 1994; Tropper and Manning 2007).